



# Standard Test Method for Estimating the Permanganate Natural Oxidant Demand of Soil and Aquifer Solids<sup>1</sup>

This standard is issued under the fixed designation D7262; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Reapproved with editorial changes in July 2016.

## 1. Scope\*

1.1 This test method covers the estimation of the permanganate natural oxidant demand (PNOD) through the determination of the quantity of potassium permanganate ( $\text{KMnO}_4$ ) that organic matter and other naturally occurring oxidizable species present in soil or aquifer solids will consume under specified conditions as a function of time. Oxidizable species may include organic constituents and oxidizable inorganic ions, such as ferrous iron and sulfides. The following test methods are included:

Test Method A—48-hour Permanganate Natural Oxidant Demand

Test Method B—Permanganate Natural Oxidant Demand Kinetics

1.2 This test method is limited by the reagents employed to a permanganate natural oxidant demand (PNOD) of 60 g  $\text{KMnO}_4$  per kg soil or aquifer solids after a period of 48 hours (Method A) or two weeks (Method B).

1.3 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice [D6026](#).

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 This standard does not purport to interpret the results of the data. It is the responsibility of the user of this standard to interpret the results obtained and to determine the applicability of these results prior to use.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D18](#) on Soil and Rock and is the direct responsibility of Subcommittee [D18.21](#) on Groundwater and Vadose Zone Investigations.

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## 2. Referenced Documents

### 2.1 *ASTM Standards*:<sup>2</sup>

[D653 Terminology Relating to Soil, Rock, and Contained Fluids](#)

[D1193 Specification for Reagent Water](#)

[D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction](#)

[D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing](#)

[D6026 Practice for Using Significant Digits in Geotechnical Data](#)

[D6051 Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities](#)

[D6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations](#)

[D6282 Guide for Direct Push Soil Sampling for Environmental Site Characterizations](#)

[D6286 Guide for Selection of Drilling Methods for Environmental Site Characterization](#)

### 2.2 *Other Standards*:<sup>3</sup>

[Method 4500— \$\text{KMnO}\_4\$  Standard Methods for the Examination of Water and Wastewater, 20th Ed. 1998](#)

[ANSI/AWWA B603-03 Standard for Permanganates](#)

## 3. Terminology

3.1 For common definitions of technical terms in this standard, refer to Terminology [D653](#).

### 3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *permanganate natural oxidant demand (PNOD)<sub>t</sub>*—the mass of potassium permanganate consumed per mass of soil or aquifer solids as a function of time.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American Water Works Association (AWWA), 6666 W. Quincy Ave., Denver, CO 80235, <http://www.awwa.org>.

\*A Summary of Changes section appears at the end of this standard

3.2.2 *maximum permanganate natural oxidant demand (PNOD<sub>max</sub>)*—the maximum mass of potassium permanganate consumed per mass of soil or aquifer solids over time.

3.2.3 *permanganate natural oxidant demand kinetics*—the rate at which potassium permanganate is consumed by soil or aquifer solids.

#### 4. Summary of Test Method

4.1 Many organic and reduced inorganic substances present in soil and aquifer solids can be oxidized by permanganate. A standard potassium permanganate solution is added to a specific amount of soil or aquifer solids and allowed to react for a period of 48 hours (Method A) or two weeks (Method B). The residual permanganate concentration is measured at prescribed sampling times and the difference in concentration is used to calculate the PNOD<sub>t</sub> at that time.

4.2 Many organic and reduced inorganic substances present in soil and aquifer solids can be oxidized by permanganate. However, some organic compounds react slowly and may not be completely oxidized within the test period while others may resist oxidation altogether.

#### 5. Significance and Use

5.1 The test method is used to estimate the permanganate natural oxidant demand exerted by the soil or aquifer solids by determining the quantity of potassium permanganate that is consumed by naturally occurring species as a function of time. Typically the measurement of PNOD is used to screen potential sites for in situ chemical oxidation (ISCO) with permanganate (Test Method A) and provide information to aid in the design of remediation systems (Test Method B).

5.2 While some oxidizable species react relatively quickly (that is, days to weeks), others react more slowly (weeks to months). Consequently, the PNOD<sub>t</sub> is expected to be some fraction of the PNOD<sub>max</sub>.

5.3 Due to mass transport related issues at the field-scale it is reasonable to assume that the PNOD<sub>t</sub> measured using the test method may overestimate the demand exerted during ISCO applications.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/and so forth.

#### 6. Interferences

6.1 Manganese oxides produced as a result of permanganate reduction may interfere with the analysis of permanganate (Method 4500-KMnO<sub>4</sub>).

#### 7. Apparatus

7.1 *Reactor Apparatus*—A 250-mL glass vial (borosilicate glass or equivalent) with an oxidant resistant screw cap is recommended. Zero headspace is not required.

7.2 *Apparatus for Drying Samples*—A laboratory oven capable of delivering sufficient controlled heat to maintain a temperature of 105°C (±10°C).

7.3 *Balance Scales*—A balance having a minimum capacity of 100 g and meeting the requirements of Guide D4753, readable (with no estimation) to 0.1 % of the test mass or better.

#### 8. Hazards

8.1 When performing laboratory analysis and handling chemicals, safety is a critical component. For this procedure, contact lenses may not be worn. Recommended personal protective equipment (PPE) for this procedure includes rubber gloves, safety glasses or goggles and a lab coat or rubber apron.

8.2 In the event of any chemical spill, refer to the specific MSDS for a proper clean-up procedure. In the case of solid potassium permanganate, sweep the solid into a clean container and dispose according to state and local regulations. A potassium permanganate spill should be diluted with water to less than 4 % strength, collected and disposed of in an approved manner. Paper or cloth towels should not be used to clean any permanganate spill.

8.3 Excess permanganate solutions can also be neutralized by sodium thiosulfate, citric acid, or other reducing agents. Solution concentrations must be less than 4 % prior to addition of any reducing agent. Excess heat can be generated and there is a potential for an unwanted reaction.<sup>4</sup>

#### 9. Sampling

9.1 Collect the sample(s) in accordance with Practices D6051, D6169, D6282, or D6286. A minimum of 600 grams of soil or aquifer solids is required from each sampling location.

9.2 Samples can be preserved at 4°C for up to 28 days. However, it is the responsibility of the users of the test method to ensure the maximum holding time for their samples.

<sup>4</sup> Additional references on general laboratory safety and procedures can be found at: <http://www.ceet.niu.edu/labs/safety.html>, <http://keats.admin.virginia.edu/lsm/home.html>, and [http://www.ehs.iupui.edu/ehs/prog\\_chemlabsafety.asp](http://www.ehs.iupui.edu/ehs/prog_chemlabsafety.asp).

### TEST METHOD A—48-HOUR PERMANGANATE NATURAL OXIDANT DEMAND

#### 10. Scope

10.1 The test method is appropriate for the determination of the 48-hour permanganate natural oxidant demand of soil and aquifer solids. The test is designed to be used for site screening purposes only. Research has shown that a large percentage of

the total permanganate natural oxidant demand can be expressed after a period of 48 hours. Consequently, this test method should not be used to determine the mass of oxidant